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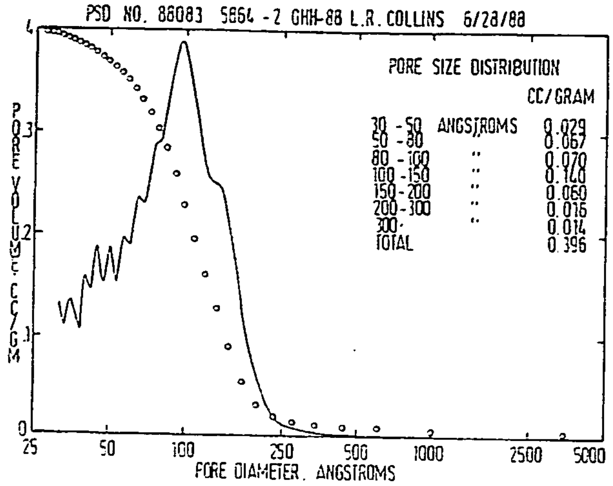
(54) Catalytic process for manufacture of low pour lubricating oils.

(57) A catalytic dewaxing process for production of low-pour lubes from bright stock. Catalyst aging is reduced, and cycle life prolonged, by use of a catalyst 60% of the pore volume of which is in less than 20 nm diameter pores. The catalyst comprises a 1-12 constraint index zeolite in a matrix which may be composed of certain forms of alumina, and optionally contains a Group VIII metal such as nickel.

EP 0 431 448 A1

FIG. 1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80 - 100 " 0.070
100 - 150 " 0.140
150 - 200 " 0.060
200 - 300 " 0.016
300 - " 0.014
TOTAL 0.396
PORE VOLUME, CC/GM
.4
.3
.2
.1
0
25
50
100
250
500
1000
2500
5000
PORE DIAMETER, ANGSTROMS

CATALYTIC PROCESS FOR MANUFACTURE OF LOW POUR LUBRICATING OILS

This invention relates to the processing of higher molecular weight vacuum distillates such as bright stock raffinates to lower their pour point.

A suitable feedstock for lubricant refining contains a quantity of lubricant stock having a predetermined set of properties such as appropriate viscosity, oxidation stability, and maintenance of fluidity at low temperatures. The process of refining to isolate that lubricant stock consists of a set of subtractive unit operations which removes the unwanted components. The most important of these unit operations include distillation, solvent refining, and dewaxing, which basically are physical separation processes in that if all the separated functions were recombined one would reconstitute the crude oil.

The complexity of the molecular makeup of the desired components is reflected by the first step in the lube refining process, vacuum distillation. In vacuum distillation, the feed is separated into four boiling fractions, each of which has a different viscosity and a different mix of aromatics, naphthenes, paraffins, resins and asphaltenes. Separation must be exact because the resulting viscosity and composition cannot be adjusted later in subsequent refining steps.

The lightest fraction of the vacuum distillation step, vacuum tower overhead, has a boiling range up to approximately 700°F (370°C) and is usually consigned to a fuel refinery to be made into light products. The remaining three fractions can be used in preparing lube stock.

The second fraction, light neutral distillate, has a boiling range between approximately 700 and 850°F (371 and 454°C), and is used to make low viscosity oils. The third fraction, heavy neutral distillate, with a boiling range of from 850 to 1000°F (454 to 538°C), is used to make high viscosity oils. The fourth fraction, vacuum resid, will not boil even under vacuum and will result in a lube base called "bright stock". This vacuum resid contains most of the resins and metals in the crude and all of the asphaltines.

Crude oils suitable for the manufacture of lubes are becoming less available due to exhaustion of reserves, and obtaining a steady, adequate supply from a known course can be difficult. Accordingly, the desirability of upgrading a crude fraction normally considered unsuitable for lubricant manufacture to one from which good yields of lubes can be obtained has long been recognized. However the complexity of the molecular constitution of lubricating oils makes it necessary to consider many variables in developing practically useful processes to production of lube stock from petroleum crude oils.

"Hydrocracking", sometimes referred to in the art as "severe hydrotreating", has been proposed to accomplish such upgrading. In this process, a suitable fraction of a poor grade crude, such as a California crude, is catalytically reacted with hydrogen under pressure. The process is complex in that some of the oil is reduced in molecular weight and made unsuitable for lubes, but concurrently a substantial fraction of the polynuclear aromatics is hydrogenated to form naphthenes and paraffins. Process conditions and choice of catalyst are selected to provide an optimal conversion of the polynuclear aromatic content of the stock, since this component degrades the viscosity index and stability of the stock. Also, in the hydrocracking process, paraffins can be isomerized, imparting good viscosity index characteristics to the final lube product. The term "hydrocracking" is employed for processes such as the foregoing; by contrast the purpose of "hydrotreating" is to stabilize the lube base stock produced by hydrocracking. Hydrocracking and hydrotreating steps may be distinguished also by the amount of hydrogen consumed, the hydrocracking step typically consuming about 178 to 356 m^3/m^3 (1000-2000 SCF/bbl) while the hydrotreating step consumes only about a tenth as much.

The hydrocracking process for increasing the availability of lube oils has an attractive feature that is not immediately apparent. Thus the composition and properties of hydrocracked stocks are generally not particularly affected by the source and nature of the crude, i.e., they tend to be much more alike than fractions prepared from different crudes by conventional means. The process therefore promises to free the refiner from dependence on a particular crude, with all of the advantages that this freedom implies.

Hydrocracked lube stocks tend to be unstable in the presence of air when exposed to sunlight. On such exposure, an unacceptable sludge is formed, sometimes very rapidly and in fairly substantial amount. Additionally, some hydrocracked lube oils tend to darken or to form a haze. Several methods have been proposed to correct this instability, such as those described in US-A-4,031,016, 3,666,657, 3,530,061, 4,162,962, 3,530,061 and 3,852,207.

Hydrocracked lubricating oils generally have an unacceptably high pour point and require dewaxing. Catalytic methods for dewaxing have been proposed, for instance in US-A-3,700,585. Hydrotreating after catalytic dewaxing is disclosed in US-A-4,137,148. US-A-4,283,271, 4,283,272, and 4,414,097 disclose processes for producing dewaxed lubricating oil base including hydrocracking a hydrocarbon feedstock, catalytically dewaxing the hydrocrackate and hydrotreating the dewaxed hydrocrackate, and employ catalyst

compositions comprising zeolite ZSM-5, ZSM-11 and ZSM-23 for the dewaxing phase.

There is a need for processes which can efficiently provide modern high quality lubricants from interchangeable and readily available low grade crudes, and for manufacturing lubricating oils, including hydrocracked lubricating oils, having a low pour point

This invention provides an energy-efficient continuous process for producing dewaxed hydrocracked lubricating oil stock from a hydrocarbon feedstock effluent boiling over about 288 °C (550 °F), by passing such an effluent through a catalytic hydrodewaxing process in which the supported catalyst composition comprises a zeolite and matrix and, optionally, a Group VIII metal.

According to the present invention a cyclic catalytic dewaxing process for improving the pour point of a demetallized petroleum fraction free of components boiling below 371 °C, comprises contacting a bright stock raffinate with a dewaxing catalyst at a temperature of 260 to 482 °C, a pressure of 14.8 to 242.4 bar, a liquid hourly space velocity of 0.2 to 20 and a hydrogen circulation rate of 89 to 3560 m^3/m^3, said catalyst comprising a porous composite of a zeolite having a constraint index of 1 to 12 and a matrix whereof the zeolite constitutes 35 to 75 weight percent, at least 60 volume percent of the pores of said composite having a diameter less than 20 nm, the composite having a particle density of at least 1.0 g/cc.

In the catalytic processing of heavy petroleum fraction it is generally believed to be desirable to have as open a catalyst pore structure as possible, i.e., a large average pore diameter while maintaining a reasonable surface area, in order to reduce diffusion limitations. Thus, for example, in petroleum resid demetallation catalysts it is desirable to minimize the number of pores with diameters less than 7.5 nm.

Unexpectedly, we have now found that in the catalytic dewaxing of heavy oil fractions such as lubricant basestocks, it is desirable to maximize the number of pores with diameters less than 20 nm in the dewaxing catalyst composite. Specifically, we find that the aging rate of the catalyst in the dewaxing reaction is affected by the catalyst pore size distribution. As will be demonstrated in the examples, the beneficial effect of catalyst pore size distribution becomes more significant as the average molecular weight of the feedstock increases.

In the drawings:

Figure 1 is a plot of pore volume (cc/gm) vs. pore diameter;

Figure 2 is a plot of reaction temperature vs. days on stream;

Figure 3 is a plot of reaction temperature vs. days on stream; and

Figure 4 is a plot of reactor temperature vs. days on stream.

In accordance with the invention the aging rate of dewaxing catalysts in dewaxing has been reduced. Accordingly, the start-of-cycle temperature in the second cycle, and subsequent cycles, of a multicycle dewaxing operation is reduced. The capacity to reduce the aging rate of the dewaxing catalyst allows for longer periods between oxygen regeneration and hydrogen reactivation cycles and thus increases overall catalyst lifetime. It is noted that aging rate can be expressed by the daily increase in operating temperature necessary to maintain a product of constant pour point. The benefits of the invention apply particularly to bright stock raffinate feedstocks, which induce among the fastest catalyst aging rates.

Physical properties such as pore volume, pore size distribution and particle density can significantly affect catalytic performance. A good example of this is in the design of demetallation catalysts, where a majority of the pores must be in the greater than 10 nm range to prevent pore plugging by deposited metals and to allow for diffusion of Ni- and V-containing porphyrins. The design of the demetallation catalysts involves a critical balance between the required pore size distribution and the high surface area needed to produce the requisite activity.

By comparison, in accordance with the invention, the catalyst is a porous mixture, at least 60 percent of the pores of which, as measured by mercury porosimetry, have diameters less than 20 nm. The average pore size of the mixture is preferably lower than 15 nm, more preferably lower than 10 nm, and in certain embodiments is less than 7 nm, and even less than 4 nm. Preferably the particle density of the porous composite is at least 1.0 g/cc.

The zeolite component of the catalyst has a constraint index of 1 to 12. The method by which constraint index is determined is described fully in US-A-4,016,218.

Constraint index (CI) values for some typical materials are:

	CI (at test temperature °C)	
ZSM-4	0.5	(316)
ZSM-5	6-8.3	(371-316)
ZSM-11	5-8.7	(371-316)
ZSM-12	2.3	(316)
ZSM-20	0.5	(371)
ZSM-22	7.3	(427)
ZSM-23	9.1	(427)
ZSM-34	50	(371)
ZSM-35	4.5	(454)
ZSM-48	3.5	(538)
ZSM-50	2.1	(427)
TMA Offretite	3.7	(316)
TEA Mordenite	0.4	(316)
Clinoptilolite	3.4	(510)
Mordenite	0.5	(316)
REY	0.4	(316)
Amorphous Silica-alumina	0.6	(538)
Dealuminized Y	0.5	(510)
Erionite	38	(316)
Zeolite Beta	0.6-2.0	(316-399)

It will be appreciated that it may be possible to so select test conditions, e.g. temperature, as to establish more than one value for the constraint index of a particular zeolite. This explains the range of constraint indices for some zeolites, such as ZSM-5, ZSM-11 and Beta.

Preferably, the crystal size of the zeolite ranges from 0.02 to 0.05 micrometers. The catalyst composition will generally, although not necessarily, contain a Group VIII metal, which when present may be exchanged into the composition, impregnated therein or physically and intimately admixed therewith by well known techniques. The amount of Group VIII metal in the catalyst composition can range from 0.10 to 10 weight percent, preferably from about 0.1 to about 3 weight percent, most preferably from about 0.2 to about 1 weight percent, based on the total weight of the catalyst composition. The Group VIII metal component can be platinum, palladium, iridium, ruthenium, cobalt, nickel, copper, molybdenum and mixtures thereof. The preferred metal is nickel. The Group VIII metal may be present alone or together with a metal from Group VI, such as chromium, molybdenum, tungsten and mixtures thereof.

The particle size (effective or hydraulic diameter) of preferred forms of the catalyst ranges from 0.75 to 3.2 mm. The binder or matrix is suitably an alumina other than alpha alumina, preferably gammaalumina. Generally it is a small particle alumina with a packing density of 0.5 to 0.6 g/cc. The matrix is characterized by a particle density of 0.95 to 1.50 g/cc. Alumina monohydrate (pseudoboehmite) is converted into a desirably dense analogue during catalyst formation.

An alumina source for the matrix used for catalyst preparation preferably has a water content of 20 to 30 weight percent. By way of illustration, the alumina monohydrate used as the source of matrix material in the Examples has a water content of 28 percent. Preferably, the alumina source is convertible to gamma-alumina. Gamma-alumina is a phase of alumina which occurs as a result of calcination at a temperature of no greater than about 538° C (1000° F). As noted in the Examples, deltaalumina is a result of calcination at a temperature of about 927° C (1700° F).

In a favoured catalyst preparation procedure the zeolite, a Group VIII metal and a source of binder or matrix are mulled together to form a homogeneous mixture. The important feature of the mulling step is the

water content of the mixture. With respect to the mixture resulting from mulling for use in extrusion, the water content can be expressed as ash content of the mixture: the ash content can range from 40 to 60 wt%. Water content can also be expressed as solid content, which can range from 45 to 60, preferably from 42 to 55 weight percent. Mulling is undertaken at a temperature ranging from 10 to 37.7 °C (50 to 100 °F). The mixture may contain 0.05 to 5 wt% of an extrusion aid such as polyvinyl alcohol.

Extrusion is undertaken in conventional apparatus to produce a particle with a length to diameter ratio of at least 2.

The matrix precursor can be alumina monohydrate (.e., pseudoboehmite), which during catalyst formation is converted to a more dense analogue.

The feed to the dewaxing unit is a fraction produced by vacuum distillation of crude oil which has been demetallized and is free of boiling range components having a boiling range up to about 700 °F, in particular one based on the vacuum resid which as noted above will produce a lube base called bright stock.

Process conditions in the catalytic dewaxer are summarized in the following Table.

DEWAXING

	Dewaxing Conditions	
Pressure, broad, bar (psig)	14.8 (200-)	242.4 (3,000)
, preferred	14.8 (200-)	140 (2,000)
Temperature, broad, °C	260-	482
, preferred	260-	375
LHSV*, broad	0.2-	20
, preferred	0.5-	5
H_2 gas, m^3/m^3 (SCF/bbl)**		
, broad	89 (500-)	3560 (20,000)
, preferred	89 (500-)	534 (3,000)

* LHSV = liquid hourly space velocity, i.e., volumes of feed per volume of catalyst per hour.

** SCF/bbl = standard cubic feet per barrel.

A preferred embodiment of the invention is described in the Examples which follow.

EXAMPLE 1

A lube dewaxing catalyst was produced by preparing a mixture of 65 percent ZSM-5, 35 percent of a coarse, high-chemical purity, low density, high-surface-area alpha alumina monohydrate (pseudoboehmite), nickel nitrate hexahydrate, and enough water to form a paste-like mixture, mulling this mixture; and extruding it into 1.6 mm (1/16 inch) cylindrical pellets by conventional means. The catalyst was pre-calcined in nitrogen for 3 hours at 482 °C (900 °F) (2.8 °C/min heat-up) and then in air at 538 °C (1000 °F) for 3 hours. This procedure converts the alpha alumina monohydrate into gamma-alumina. The physical properties and pore size distribution (as measured by mercury porosimetry) are shown in Table 1. The catalyst in this example will be referred to as a medium pore diameter catalyst. This catalyst has 60 percent of its pore with diameters less than 20 nm (as measured by mercury porosimetry).

EXAMPLE 2

A lube dewaxing catalyst was produced by preparing a mixture of 65 percent ZSM-5, 21 percent of the same alpha alumina monohydrate (pseudoboehmite) used in Example 1, 14 percent delta alumina, nickel

nitrate hexahydrate, and enough water to form a paste-like mixture, mulling this mixture, and extruding it into 1.6 mm (1/16 inch) cylindrical pellets by conventional means. The delta alumina was prepared by calcining the alpha alumina monohydrate in air at 927 °C (1700 °F) for 4 hours. The catalyst was precalcined in nitrogen for 3 hours at 482 °C (900 °F) (2.8 °C/min heat-up) and then in air at 538 °C (1000 °F) for 3 hours. The physical properties and pore size distribution (as measured by mercury porosimetry) are shown in Table 1. The catalyst in this example will be referred to as a large pore diameter catalyst. This catalyst has 44 volume percent of its pores (as measured by mercury porosimetry) with diameters less than 20 nm.

EXAMPLE 3

A lube dewaxing catalyst was produced by preparing a mixture of 65% ZSM-5, 35% of a finely divided, dense, high chemical purity alpha alumina monohydrate, Keith alumina (pseudoboehmite from Keith Corporation), nickel nitrate hexahydrate, and enough water to form a paste-like mixture, mulling this mixture, and extruding it into 1.6 mm (1/16 inch) cylindrical pellets by conventional means. The catalyst was precalcined in nitrogen for 3 hours at 482 °C (900 °F) (2.8 °C/min heat-up) and then in air at 5.38 °C (1000 °F) for 3 hours. This procedure converts the alpha alumina monohydrate into gamma alumina. The physical properties and pore size distribution (as measured by mercury porosimetry) are shown in the following Table. The catalyst in this example will be referred to as a small pore diameter catalyst. It has an average pore diameter of 4 nm, 92 volume percent of its pores (as measured by mercury porosimetry) being of diameter less than 20 nm.

Table 1

	Example 1	Example 2	Example 3
Ni, wt %	0.95	0.98	about 1.0
Na, ppm	155	140	430
Density, g/cc			
Real	2.616	2.229	2.616
Particle	0.992	0.896	1.407
Packed	0.60	0.56	.71
Pore Volume (BET), cc/g	0.626	0.777	0.328
Surface area, m^2/g	358	326	313
Pore Volume Distribution, x 10 cc/g			
30-50	0.46	0.52	0.29
50-80	1.26	0.69	0.67
80-100	0.54	0.31	0.70
100-150	0.63	0.62	1.40
150-200	0.27	0.48	0.60
200-300	0.26	0.47	0.16
300+	1.76	2.90	0.14

EXAMPLE 4

The catalysts of Examples 1 and 2 were examined for dewaxing of paraffinic North Sea Heavy Neutral and Bright Stock raffinates. Properties of the feedstocks are given in Table 2. Both catalysts were able to produce -6.6°C (20°F) pour point products. The aging rates of the catalysts were tracked by adjusting the reaction temperature to compensate for activity loss so as to maintain a target -6.6°C (20°F) pour point while maintaining all other conditions constant.

In comparing the performances of the medium and large pore diameter catalysts, only a slight aging rate benefit was observed with the medium pore diameter catalyst in dewaxing the Heavy Neutral raffinate (Figure 2).

However, a much more significant aging rate benefit was observed with this same catalyst in dewaxing the Bright Stock raffinate (Figure 3). Thus the catalyst with the smaller pore diameter is more suitable for processing the heavier feedstock.

Table 2

Properties of the Statfjord Heavy Neutral and Bright Stock Raffinates Used in this Study

	Heavy Neutral	Bright Stock
Pour Point, °C (°F)	48.8 (120)	48.8 (120)
K.V. 0 100°C, cs	11.550	27.500
K.V. 0 300°C, cs	4.550	8.890
Refractive Index 0 70°C	1.462	1.471
Gravity, API	30.700	27.500
Hydrogen, wt%	13.640	13.490
Nitrogen, ppm	110.000	420.000
Basic Nitrogen, ppm	97.000	253.000
Sulfur, wt%	0.200	0.380
Aliphatic Sulfur, wt%	0.130	0.230
Ni, ppm	0.200	0.400
V, ppm	0.100	0.100
Fe, ppm	0.250	0.350
Cu, ppm	0.200	-
Water, Azeo Dist, ppm	67.000	39.000
Color, ASTM oil	1.750	5.000
Furfural, ppm	2.000	7.000
Carbon Res, MCRT, wt%	0.030	0.470
Total Acid No.	0.280	0.200
Flash Point, Cleve Open	512.000	561.000
Molecular Weight, avg	572.000	745.000
Extr. in Pet Waxes, wt%	79.570	75.340

Distillation, (vol% Distilled)	IBP, °C (°F)	404 (759.5)	412 (774.8)
	5 vol%	473 (883.0)	511 (951.2)
	10	486 (906.0)	539 (1003.1)
	20	498 (929.3)	557 (1035.1)
	30	507 (943.7)	567 (1052.7)
	40	512 (953.7)	578 (1072.4)
	50	517 (962.3)	588 (1091.3)
	60	522 (971.9)	599 (1109.8)
	70	528 (982.4)	
	80	534 (993.9)	
	90	544 (1012.4)	
	95	554 (1029.9)	
	EP	560 (1039.8)	602 (1115.2)
	% Recov.	100	62

EXAMPLE 5

The performance of the medium pore and small pore dewaxing catalysts in the dewaxing of Statfjord Bright stock are shown in Figure 4. The process conditions were: LHSV = 0.5, 28.6 bar (400 psig) and 445 m^3/m^3 hydrogen/oil (2500 SCF H_2/bbl). In dewaxing bright stock raffinate, this example shows that the small pore Keith alumina based catalyst has an improved performance over the medium pore catalyst.

Claims

1. A cyclic catalytic dewaxing process for improving the pour point of a demetallized petroleum fraction free of components boiling below 371 °C, which comprises contacting a bright stock raffinate with a dewaxing catalyst at a temperature of 260 to 482 °C, a pressure of 14.8 to 242.4 bar, a liquid hourly space velocity of 0.2 to 20 and a hydrogen circulation rate of 89 to 3560 m^3/m^3, said catalyst comprising a porous composite of a zeolite having a constraint index of 1 to 12 and a matrix whereof the zeolite constitutes 35 to 75 weight percent, at least 60 volume percent of the pores of said composite having a diameter less than 20 nm, the composite having a particle density of at least 1.0 g/cc.

2. A process according to claim 1 wherein said zeolite is ZSM-5.

3. A process according to claim 1 or claim 2 wherein said catalyst further comprises 0.01 to 10 percent, by weight of the composite, of a Group VIII metal.

4. A process according to claim 3 wherein said metal is nickel.

5. A process according to any preceding claim wherein said matrix comprises small-particle alumina having a packing density of 0.5 to 0.6 g/cc and a particle density of 0.95 to 1.50 g/cc.

6. A process according to any preceding claim wherein the average pore diameter of said composite is less than 15 nm.

7. A process according to any preceding claim wherein the average pore diameter of the said composite is less than 10 nm.

8. A process according to any preceding claim wherein the average pore diameter of the said composite is less than 7 nm.

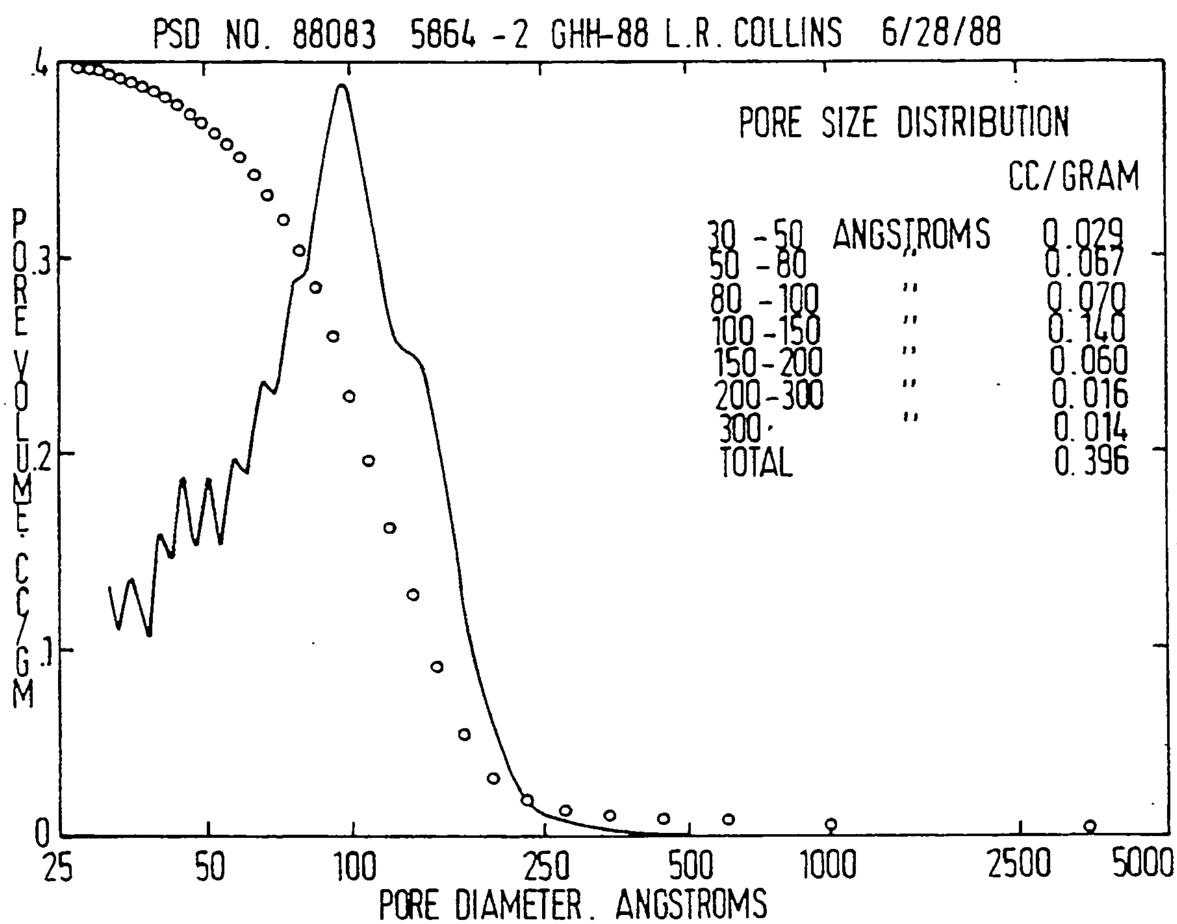
9. A process according to any preceding claim wherein the average pore diameter of the said composite is less than 4 nm.

10. A process according to any preceding claim wherein the crystal size of the zeolite is from 0.02 to 0.05 micrometers.

11. A process according to any preceding claim wherein the pour point of the product of the dewaxing is at least 39 °C (70 °F) lower than that of said bright stock.

12. A process according to any preceding claim wherein said contacting is carried out at a temperature below 375 °C, a pressure below 140 bar, a hydrogen circulation rate below 534 m^3/m^3 and a liquid hourly space velocity of 0.5 to 5.

FIG. 1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80 - 100 " 0.070
100 - 150 " 0.140
150 - 200 " 0.060
200 - 300 " 0.016
300 · " 0.014
TOTAL 0.396
PORE VOLUME. CC/GM
.4
.3
.2
.1
0
25
50
100
250
500
1000
2500
5000
PORE DIAMETER. ANGSTROMS

FIG. 2

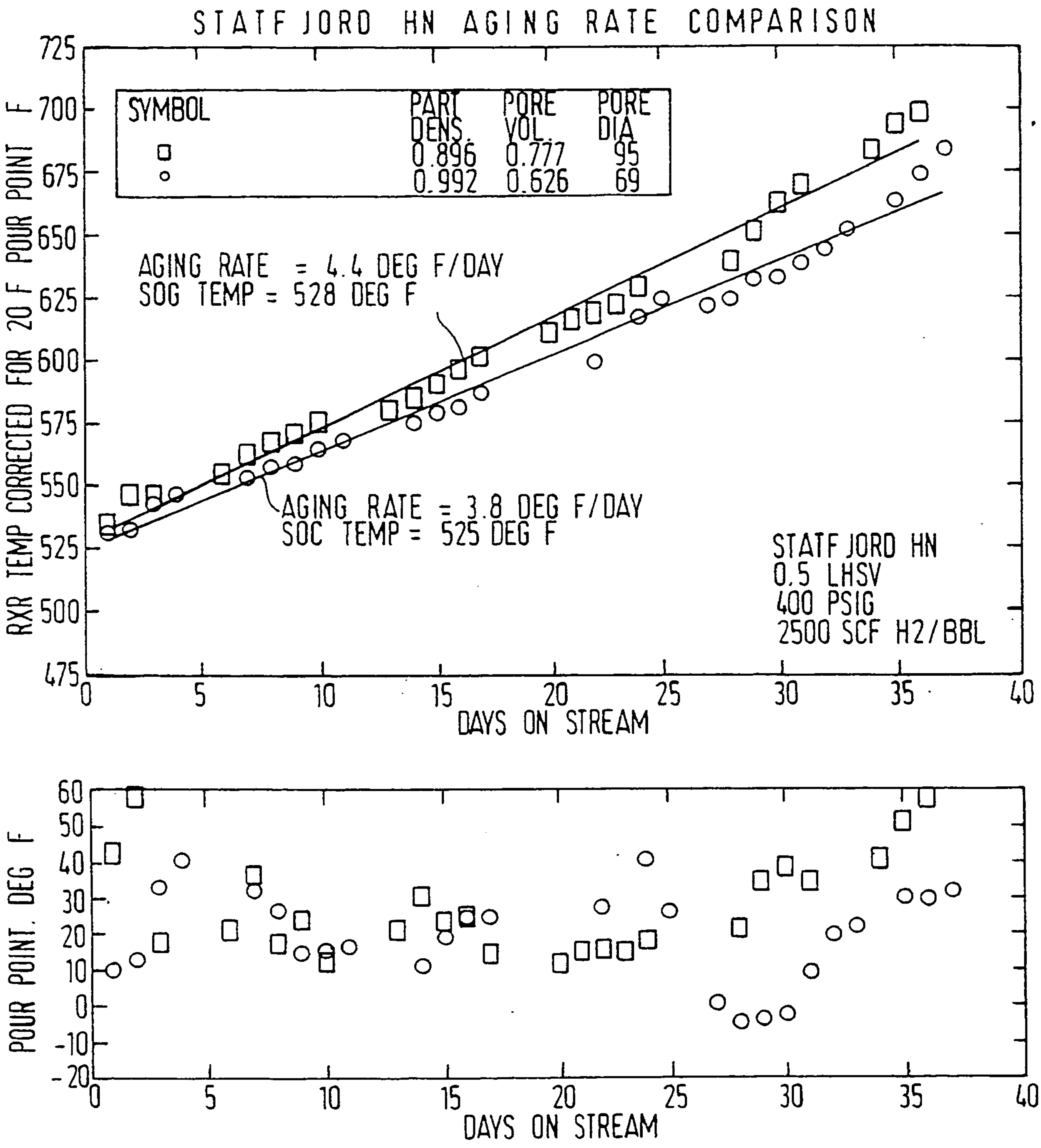
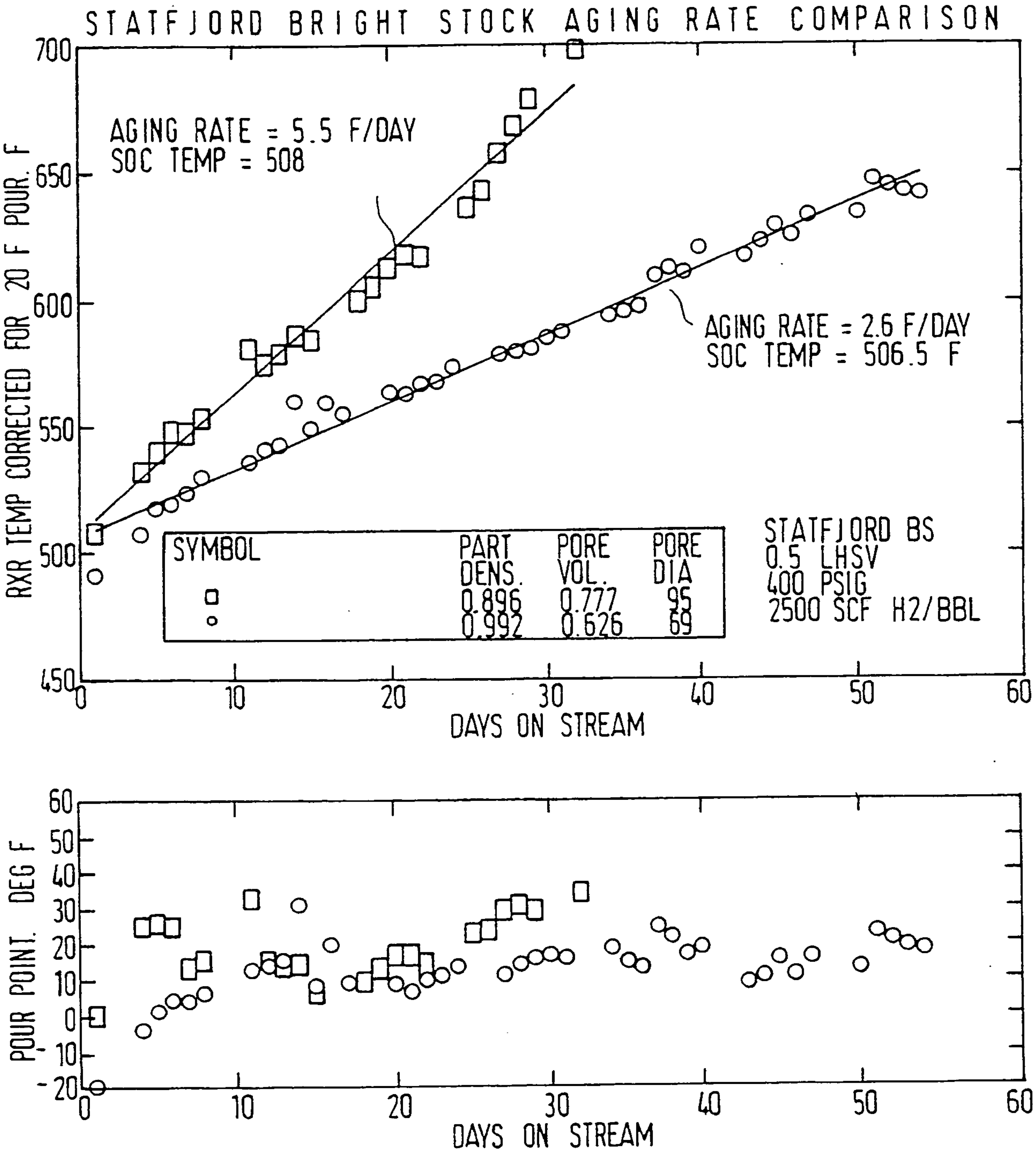

STATFJORD HN AGING RATE COMPARISON
SYMBOL
PART DENS.
PORE VOL.
PORE DIA
0.896 0.777 95
0.992 0.626 69
AGING RATE = 4.4 DEG F/DAY
SOG TEMP = 528 DEG F
AGING RATE = 3.8 DEG F/DAY
SOC TEMP = 525 DEG F
STATFJORD HN
0.5 LHSV
400 PSIG
2500 SCF H2/BBL
RXR TEMP CORRECTED FOR 20 F POUR POINT F
DAYS ON STREAM
POUR POINT. DEG F
DAYS ON STREAM

FIG. 3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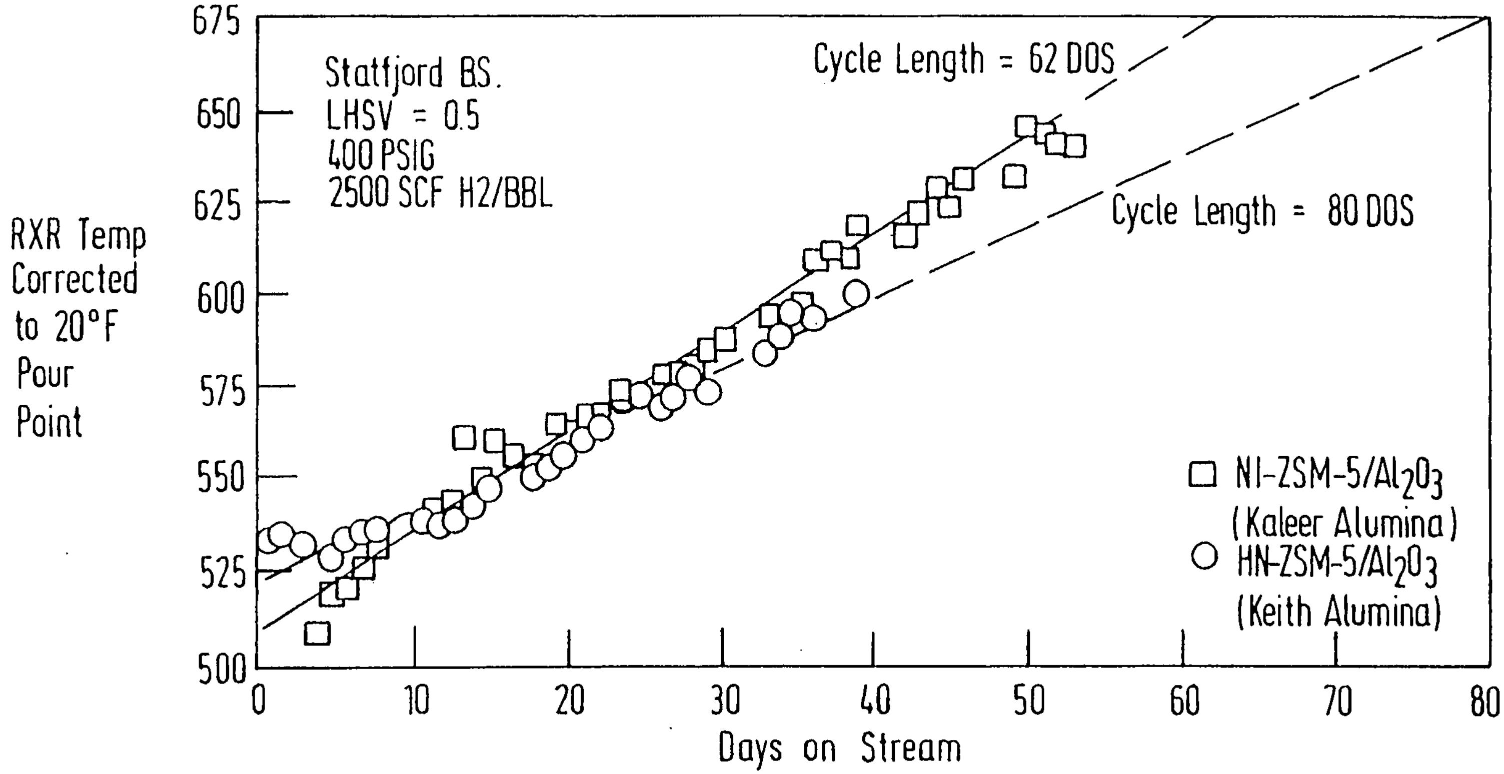
SOC TEMP = 508
AGING RATE = 2.6 F/DAY
SOC TEMP = 506.5 F
SYMBOL
PART DENS.
PORE VOL.
PORE DIA
0.896
0.777
95
0.992
0.626
69
STATFJORD BS
0.5 LHSV
400 PSIG
2500 SCF H2/BBL
RXR TEMP CORRECTED FOR 20 F POUR. F
DAYS ON STREAM
700
650
600
550
500
450
0
10
20
30
40
50
60
POUR POINT. DEG F
DAYS ON STREAM

FIG. 4

Alumina Bound ZSM-5
Dewaxing of Statfjord Bright Stock



European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 90 12 2676

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
A	US-A-4 508 615 (OLECK et al.) * Claims 1-12; examples 4,5,6 * ---	1,2,3,4,5,6,11	C 10 G 45/64
A	EP-A-0 043 681 (MOBIL) * Claims 1-6; page 4, paragraph 3 and last paragraph * -----	1,2,3,4	

TECHNICAL FIELDS SEARCHED (Int. Cl.5)

C 10 G

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	11-02-1991	DE HERDT O.C.E.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

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